IMPROVED RESEALING PROCEDURES FOR THE SECOND DESEAL/RESEAL PROGRAM IN RAAF F111 AIRCRAFT FUEL TANKS

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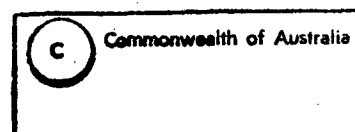
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Improved Resealing Procedures for the Second Deseal/Reseal Program in RAAF F111 Aircraft Fuel Tanks

S. Batten, R.H.E. Huang and L.V. Wake

Abstract

A high temperature-resistant polyester sealant was originally employed to seal faying surface grooves in F111 fuselage fuel tanks as well as structural voids and aerodynamic surfaces. Chemical hydrolysis of this material was detected in March 1974 and led to its replacement by a polysulfide sealant in the late 70's and early 80's. Where possible, the polyester was removed, however much of it remained inaccessibly located between the overlapping panels. It was found that the continued hydrolysis of this residual polyester generated sufficient pressure to penetrate the polysulfide sealant resulting in fuel leaks as well as appearing externally in trails along the skin of the aircraft. To protect the polysulfide from penetration, a mechanical barrier was applied in the form of a layer of epoxy polyamide between the polysulfide and the polyester. This system was successful for some years, however the occurrence of fuel leaks had become unacceptable by 1987. All F111 aircraft suffered numerous fuel leaks over the period 1988-91 including a number of safety-of-flight (SOF) incidents. A detailed investigation was therefore requested to identify the cause(s) of sealant failures. This investigation found that the sealant and the underlying epoxy barrier coating could be manually peeled from the painted tank surface when prepared by the recommended methods and that two procedures were responsible for the poor adhesion. One of the procedures involved application of the barrier coating over incompletely cured priming paint resulting in solvent attack of the barrier. The second procedure, involving the use of a so-called "titanate adhesion promoter", significantly reduced peel strength of the barrier coating to the paint. Modification of these two steps resulted in adhesive bond strengths greater than the cohesive strength of the polysulfide sealant. These changes were adopted for the second deseal/reseal program of the F111 aircraft fleet. Following teething problems with the first aircraft, the procedures were improved and the F111 fleet remains leak-free after two years.

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Authors

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Stuart Batten undertook work on aspects of the present report as a vacation scholar during the period December 1990 - January 1991. Stuart Batten, who was a 1st Class Honours Student at the time of this work is currently undertaking a PhD at the University of Melbourne majoring in inorganic chemistry.

R.H.E. Huang

Robert Huang holds a BSc degree from the University of Adelaide, South Australia. After two and a half years of experience in Rubber Technology with the Olympic Tyre and Rubber Company, Melbourne, he joined the rubber section of MRL in November 1970. Since 1975 he has been involved in R & D work on sealants as an SPOC.

L.V. Wake

Lindsay Vernon Wake was awarded a Department of Supply scholarship to complete a PhD at the University of NSW in 1969. He joined MRL in 1972 and was briefly associated with the Textiles Group and later the Marine Environment Group of MRL. Since 1981, Dr Wake has been located in Paint Technology where he has formed a close working association with the RAAF. He is currently investigating the synthesis and performance of fire retardant paints, high temperature resistant coatings, NIR camouflage paints and other coating materials of Defence interest.

Contents

Improved Resealing Procedures for the Second Deseal/Reseal Program in RAAF F111 Aircraft Fuel Tanks

1. Introduction

The fuel capacity of F111 aircraft, as in other modern aircraft, is achieved through the concept of integral fuel tanks in which use is made of internal spaces in the fuselage, wings and vertical fins (stabilisers). This concept requires the sealing of cavities, rivets and seams with flexible sealants which are required to resist turbulence, absorb shock differentials, provide corrosion protection, tolerate all weather conditions and retain fuel. The first sealants meeting these requirements were based on polysulfide materials cured by calcium dichromate. Later, manganese dioxide cured polysulfide systems were introduced with specific gravity modifications.

Two types of sealants were initially applied to the integral fuel tanks of F111 aircraft, these being (i) a polysulfide sealant applied as an internal fillet and on seams and fasteners within tanks and (ii) a polyester sealant applied between faying, i.e. overlapping, surfaces and externally in grooves and voids as an aerodynamic filler (Figure 1).

The polysulfide sealant (i) was applied in a two-stage process involving the initial brush application of thin coats of solvent-thinned polysulfide followed by the application of a fillet of solventless polysulfide. It has been found that brush application of thinned sealant provides good adhesion to the substrate whereas the fillet of solventless polysulfide provides good fuel sealing properties.

The polyester sealant (ii) was originally selected for use because of its excellent high temperature properties. However, its susceptibility to hydrolytic breakdown was never examined and, as a result of this shortcoming, extensive fuel leaks occurred in RAAF and USAF aircraft after a relatively short period of time. As a consequence of the polyester breakdown, all RAAF F111 aircraft underwent an extensive deseal/reseal program between 1977 and 1982. To augment the resistance of the polysulfide sealant to penetration caused by expansion of the hydrolysing polyester [1,2], a relatively rigid epoxy polyamide material was applied across all gaps as a supplementary barrier. Polysulfide was then applied over the epoxy barrier coating to protect it from aviation turbine fuel and fuel additives.

GENERAL SEALANT
(FMS-1044)

FUEL AREA

CORROSION
PROTECTION
COATING

FAYING SURFACES

SEALING GROOVES

FILLET SEALANT
(FMS-1043)

FASTENER
INSTALLATION

STRUCTURAL VOIDS

AERODYNAMIC
SMOOTHER

NON-FUEL AREA

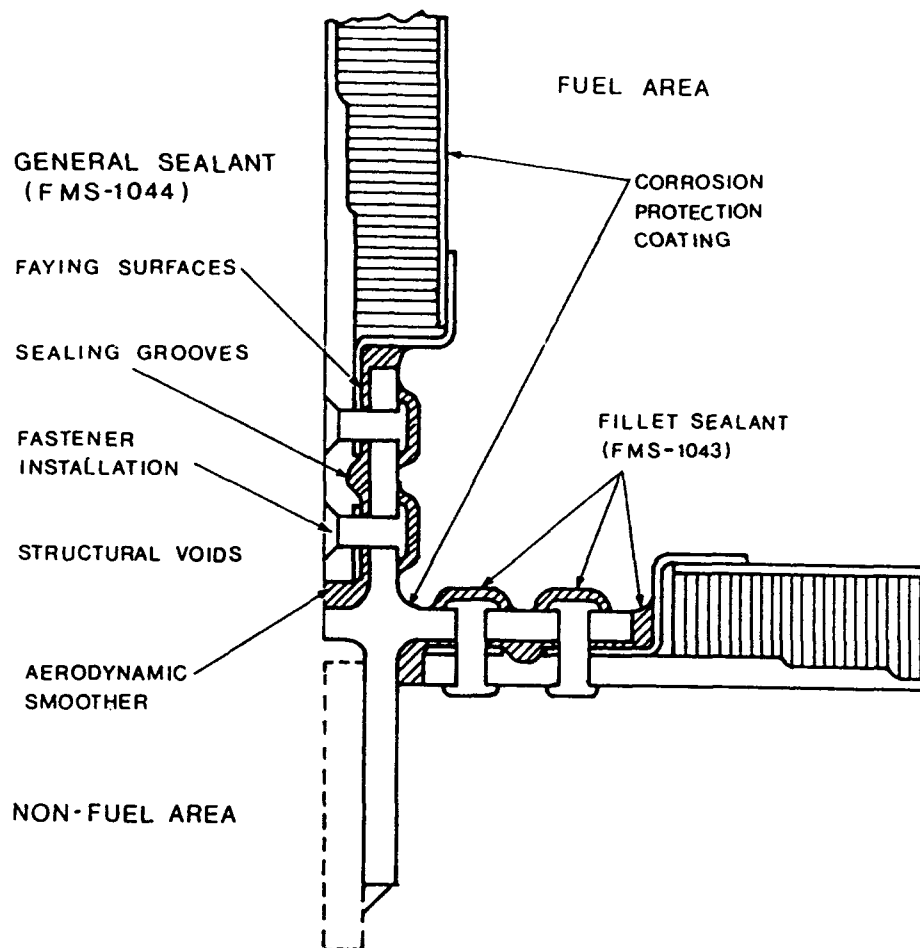*Figure 1: Original sealant system employed in F111 fuselage fuel tanks.*

From the time of the first deseal/reseal program until adoption of the present
findings for the second deseal/reseal program, resealing of the F111 fuel tanks has
involved:

(i)   repainting of integral fuel tank surfaces where sealant is to be applied and
        curing the paint for three days at ambient temperatures,

(ii)   application of a solution of a so-called `titanate adhesion promoter' over the paint, drying for 30 minutes, and removing adhesion promoter residues with a solvent,

(iii)   filling all gaps between mating surfaces with a thin continuous bead of epoxy barrier coating and curing the epoxy for 24 hours,

(iv)   reapplication of adhesion promoter, drying and removing residues (as in (ii)),

(v)   application of two layers of solvent thinned polysulfide and two layers of solventless polysulfide sealant over the epoxy barrier coating and across adjacent painted surfaces to a width of approximately 3 cm.

This repair system has worked to a limited degree, however reports of fuel leaks were received by the mid-eighties. The severity of the problem gradually increased (Tables 1 & 2) to the extent that a decision was made to undertake a second deseal/reseal program. In view of this decision, RAAF requested MRL to review F111 tank sealing procedures [3] for possible improvements to long term durability. The present report summarises the results of this investigation and of changes in RAAF procedures adopted for the second F111 deseal/reseal program.

2. *Sealant Failures in RAAF F111 Aircraft*

RAAF defect reports have generally indicated the number of leaks, the tanks involved, operational concerns, problems encountered and a discussion of the possible causes of the leak [4, 5]. Difficulty in locating the cause of a fuel leak is frequently experienced as the point where the fuel appears externally may be some distance from its source within the tank. This difficulty increases the time involved and cost of repairs [6], e.g. a fuel leak from the aft glove area of the F2 fuel tank just forward of the rotating glove above the L/H engine intake of F111 aircraft A8-132 required 608 manhours ($19,200) to be expended for location and repair of the fuel leak.

The incidence of fuel leaks has gradually increased since the first deseal/reseal to the extent that multiple leaks may be present in each aircraft [7], e.g. preservicing leak checks carried out on aircraft A8-109 revealed over 94 fuel leaks on the fuselage and wings including potential SOF (safety-of-flight) problems with leaks into enclosed space of the aircraft backbone channel. Detailed internal (visual) inspection of most fuselage fuel tanks revealed "areas of sealant reversion[1]", badly applied sealant, many attempted patch repairs, as well as areas where sealant had not been applied at all. The unit recommended that in view of the flight safety ramifications of some of the leaks found on this aircraft, a fuel leak survey of F111 fleet should be undertaken to assess the need for any

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[1] The term reversion has commonly, but erroneously, been used to describe the sealant degradation process. The cause of the degradation was hydrolysis which is different to reversion.

acceleration of the deseal programme for the remaining wings and for the possible commencement of a programme of fuselage deseals.

RAAF Headquarters Logistic Command (HQLC) began requesting reports of all fuel leaks in F111 fuel tank by 1987. The accumulated records show that all operational aircraft suffered fuselage sealant failures between the period 1988-91 [8]. Some of the failures involved poor workmanship such as inclusion of voids or insufficient sealant application, however many failures were observed where materials and procedures appeared to have been followed correctly.

Table 1: F111 Fuselage Fuel Tank Summary - as at June 91

AIRCRAFT NO	1988	1989	1990	1991
A8-109	6	33	DS/RS	10
A8-112	6	4	DS/RS	1
A8-113	8	8	DS/RS	DS/RS
A8-114	5	4	Pt DS/RS	2
A8-125	NR	4	6	48
A8-126	17	NR	15	DS/RS
A8-127	3	5	16	DS/RS
A8-129	NR	5	22	NIL
A8-130	3	9	10	NIL
A8-131	NR	8	DS/RS	NIL
A8-132	NR	NR	7	DS/RS
A8-134	7	17	R5	NIL
A8-135	2	7	18	NIL
A8-138	4	3	R5	NIL
A8-140	3	8	30	NR
A8-142	2	NR	5	NIL
A8-143	NR	2	R5	NIL
A8-144	NR	NR	2	NIL
A8-145	NR	NR	2	NIL
A8-146	NR	NR	38	NIL
A8-147	NR	6	1	1
A8-148	1	NR	NR	NIL

DS/RS	=	Undergoing deseal/reseal program
Pt DS/RS	=	Partly desealed/resealed
NR	=	not recorded.
R5	=	aircraft service

Table 2: F111 Fuselage Fuel Leak Summary - as at June 91

AIRCRAFT	1988	1989	1990	1991
A8-109 De/reseal DEC76 USA	T, Upper S,R,1 S,L,1,4 S,R,3 A1,R,4	F2,L*8 F2,R*1 F1,L,1*2 T,R,1*2 A1/A,2*13 S,L,1*5 S,R,1*3	Aircraft underwent part DS/RS at 3AD/482 May 89-Oct 90	F2,L,2,3*3 F2,R,2,3*2 T, Lower*1 S,R,2*1 S,L,2*1 A1,L,2*2 DS/RS scheduled
A8-112 De/reseal SEP78 USA NOV90 USA		S,R,1 F2,R,1*3 F2,L,1*2	F2,R,1,1-3 A1,L,1 Underwent DS/RS USA	T, lower Work in this area
A8-113 De/reseal OCT77 USA	A1,R,1-4 A1,L,1-4	A1,R,1-4 A1,R,1-4	R5 completed. undergoing DS/RS at SMALC	DS/RS completed in JUN91
A8-114 De/reseal NOV77 USA	F1,L,1,2 F1,R,2	A1,R,2*2 A1,L,2*2	Aircraft had DS/RS on A1, A2, S tanks at 3AD89/90	A1,L,2 DS/RS NOV91
A8-125 De/reseal MAR80	F2,R,*4 F2,L,*2 A1/A2*13	A1,R,4*3 A2,L,1*3		F2,R,1*2 S,L,3*3,4*6 S,R,1*5,2*6
A-8-125				S,R,3*5,4*6 A2,L,2*4,3*4 A2,L,4*2 A2,R,1*2,3*3 A2,R,4
A8-126	F1,R,3,4 F2,R,1,2,3 F2,L,1,2 S,R,2,3 A1,R,4 A1,L,2,4 A2,R,1 A2,L,1,4		F2,L,1,2,3 A1,L,3 A1,R,2 A2,L,3 A2,R,1,3,4 S,L,1,2,3,4 S,R,3	Undergoing DS/RS AMB by H de H

Table 2 (Contd): F111 Fuselage Fuel Leak Summary - as at June 91

AIRCRAFT	1988	1989	1990	1991
A8-127				
	F2	F2,R*3	F1,L,4	
De/reseal	R,1,2,3	F2,L,1,3	F2,R,2*2	
MAR80		S,R,1	F2,R,3*7	
		S,L,1	F2,L,1	
			S,L,2*3	
			S,L,3	
A8-129				
	F2,R,3	F2,R*3	A1,R,2	DS/RS at
De/reseal	F2,L,1*2	A2,R,1	A2,R,2	SMALC
MAY79		S,R,1*2	F2,L,3	JUN91
		S,L,1*3	F2,R,2*2	
			F2,R,3	
			S,L,3,4,	
			S,R,3,4	
A8-130				
		A1,R,1,2	F2,L,2	
		A2,L,1,2	F2,R,1*3	
		S,L 1	F2,R,2*3	
			A1,L,2*3	
			A1,L,3*2	

FWD, SIDE, BAY............e.g. F2, R, 3
AFT, SIDE, BAY.............e.g. A2, L, 2
SADDLE, SIDE, BAY.....e.g. S, R, 1
*.............. ....Multiply

During aircraft overhaul, personnel involved with deseal/reseal occasionally observed the appearance of small protrusions of reverted polyester sealant through the polysulfide sealant. The mechanism by which the degrading polyester overcame the rigid epoxy barrier coating to reach the polysulfide is unclear but the pressure of the reverting sealant [1,2] has presumably exceeded the paint/epoxy bond strength thereby lifting the barrier coating from the paint. The degraded polyester then presumably migrated through this separation to penetrate the overlying sealant.

Discussions with RAAF personnel involved in deseal/reseal of F111 aircraft have provided some support for the suggestion that degrading sealant overcomes the barrier coating by this mechanism. They described how long strips of sealant which failed could be manually pulled from the seams, separating by adhesive delamination of the barrier coating and the paint. While the adhesion between the paint and the barrier coating is weak, the bond between the barrier coating and the relatively soft sealant has generally remained intact during service so that the sealing material separating from the tank has been a strip of combined barrier coating/sealant.

3. Experimental

3.1 Materials

3.1.1 Paint

The paint employed throughout this trial was polyurethane MIL-C-27725B
Type II, Class B, DeSoto Inc., curing solution/base/solvent reducer 823-707/916-
702/020-707. Although referred to as a polyurethane, the paint comprises a long
chain epoxy, cured with an isocyanate. The paint was mixed in the ratio of 4/1/4
of curing solution/base/solvent reducer and let stand for 30 minutes prior to
spray application. The panels were spray applied and cured for the specified
periods prior to application of the epoxy barrier coating.

3.1.2 Barrier Coatings

Of the 3M barrier coatings used in the F111 aircraft, two batches were trialled. The
current barrier coating, EC-3598 was compared to a two-year old sample of the
EC-2216 which was formerly used. EC-3598 was a high viscosity version of EC-
2216.

EC-2216, 3M Co., Lot No. 31M6R/93L6R (7parts by weight of A/5 parts by
weight B);

EC-3598, 3M Co., Lot No. 96K4/4A5 (150 pts. A/100 pts B v/v); 100 parts base
(B): 140 parts accelerator (A) Scotch-Weld.

Prior to application of the barrier coatings, EC-2216 or EC-3598, the paints were
solvent-cleaned with MIL-C-38736, a solvent mixture composed of aromatic
naphtha 50%, ethyl acetate 20%, methyl ethyl ketone 20% and isopropyl alcohol
10% by volume. One group of paints was treated with the recommended adhesion
promoter, PR-148 (Products Research Corporation), a formulation understood to
be based on a 1% solution of an alkoxy titanate in the MIL cleaner. For the peel
test, the barrier coatings were applied with a Gardner knife at an average
thickness of 2 mm and cured for 24 hours at 25 °C prior to application of the
sealant. For the lap shear tests, the barrier coating was applied to a thickness of
0.33 mm.

Alternative candidate barrier coatings examined were:

(a)   Polyamide (PA); 139 parts Versamid 253, Henkel (Aust); 100 parts E9-110,
        Epirez Aust. Pty Ltd.
(b)   Cycloaliphatic amine (CAA); 90 parts; Versamine C31, Henkel (Aust), 100
        parts E9-110, Epirez Aust. Pty Ltd.
(c)   Polyurethane (PU); 20 parts Desmophen 1100; 80 parts Desmophen 550U;
        160 parts Desmodur L67, Bayer (Aust).
(d)   Epoxy/polysulfide (EP/PS); 100 parts Capcure 3800, Henkel (Aust); 100
        parts E9-110; Epirez Aust. Pty Ltd.

Alternative candidate coatings to the 3M materials were only examined by the lap shear test.

3.1.3 Sealants

Before application of the sealant, the epoxy barrier coating was lightly abraded, wiped with the MIL-C-38736 cleaner and treated with PR-148. The Class A sealant was then applied. The Class B was mixed and applied over the Class A material after 6 hours. The sealants employed were:

PR-1750 A-2 MIL-S-83430A AMD I,
PR-1750 B-2 MIL-S-83430A AMD I.

3.1.4 Aluminium Panels

All test panels were of 2024-T81 aluminium which had been deoxidised and pretreated with a chromate conversion coating prior to painting. The side to be painted was cleaned with MIL-C-38736 and painted as per 3.1.1.

3.1.5 Adhesion Promoter

The adhesion promoter, PR-148, was freshly formulated from a concentrated solution immediately prior to application of the barrier coating or sealant. Formulation of the adhesion promoter was to a 1% v/v solution in MIL-C-38736 solvent. Adhesion promoter was then applied to the surface and allowed to dry for 30 minutes after which time residual adhesion promoter was wiped from the surface.

3.2 Examinations

3.2.1 Modified Peel Test

The modified peel (and lap shear) tests were carried out using an Instron Model 1026 instrument with a cross-head speed of 50 mm/min and a full scale load of 200 N.

The peel test was modified as follows. A piece of tape was applied across one end of a painted panel to produce a non-adhering band of barrier coating/sealant for the jaws of the Instron machine. The epoxy barrier coating was then applied to the paint and cured for 24 hours (with or without adhesion promoter) prior to application of sealant. During application, wire mesh was applied into the sealant to increase its cohesive strength. Prior to testing, the 2.5 mm wide non-adhering strip of barrier coating/sealant and the panel with a narrow cut through the epoxy, were gripped and the peel strength measured (see Figure below). Provided that the epoxy barrier coating was kept relatively thin (approximately 0.5 mm thick), the combined epoxy-sealant on the paint was sufficiently flexible to successfully measure peel strength. This modified peel test provided a method to observe whether (i) adhesive delamination would occur between the paint and the

barrier coating, (ii) adhesive delamination would occur between the barrier coating and the sealant, (iii) cohesive failure would occur within the sealant.
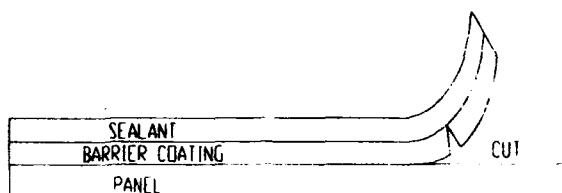
3.2.2 Modified Lap Shear Test

Barrier coating was applied to appropriate areas of pairs of painted panels and each pair was then bonded (by the drying barrier coatings) by the application of 10 kg weight for 24 hours to produce an overlap of 12 mm. Some of the painted panels were treated with adhesion promoter prior to application of the barrier coatings. The panels were cut into five separate strips, 2.54 cm wide, and immersed in fuel for appropriate periods of time. Lap shear tests were also carried out with paints which had an accelerated cure involving 24 hours at room temperature followed by 24 hours at 60 °C.

The panels were tested on the Instron using a crosshead speed of 0.5 mm/min and a full scale load of 10 kN. Each panel was also inspected to determine the mode of failure in the test.

3.2.3 Fuel Immersion of Barrier Coatings

The barrier coatings were immersed at 25°C in (i) standard F35 (Avtur fuel) and (ii) F35 (fuel with the fuel system icing inhibitor (FSII) diethylene glycol monomethyl ether at a concentration of 0.15% v/v). The effects of fuel immersion on the sealant system were monitored through percentage weight increase and percentage volume swell (by ASTM 471).

4. Results

4.1 Techniques for Determining F111 Sealant System Performance

The initial difficulty in determining sealant performance was to devise a technique to measure the strength of adhesive or cohesive failure in a sealant system consisting of three layers viz, paint/epoxy barrier/sealant. (Adhesive failures occur by failure at the interface between two materials whereas cohesive failures occur when the bonds between materials are stronger than the cohesive strength of the weakest component of the system.) A number of measurement techniques

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were examined and modified peel and lap shear techniques devised (Sections 3.2.1 and 3.2.2). The peel test was central to much of the work in that it provided a reproducible method for comparing adhesion at the interfaces against the cohesive strength of the sealant which is the weakest material.

In practice, the modified peel test generally produced failures either by adhesive separation at the paint-epoxy barrier coating interface if adhesion was poor or by cohesive failure within the sealant if adhesion between paint/barrier coating was good. The lap shear test results were consistent with those of the peel test and provided confirmation of the mode of failure. These techniques were used to determine the effect of adhesion promoter, of barrier coating composition and of cure schedules on the strength of the total sealing system.

4.2 Effect of Sealant Procedures on Adhesion and Cohesion of Sealant System

4.2.1 Effect of Paint Cure on Peel and Lap Shear Strength

Adhesion of the priming paint/3M barrier coating/sealant system increased with paint cure time up to two weeks and resulted in increasing cohesive failure (and decreasing adhesive failure) within the sealant system. Increased paint cure temperatures similarly improved adhesion as well as reducing the time required to achieve cohesive failure (Tables 3, 4: Figure 2).

For shorter paint cure times, adhesive failures (by peel test) allowed the barrier coating/sealant to be manually peeled from the paint in the manner described by RAAF operators for in-service failures. Microscopic examination of the failed lower surface of the barrier coating (Plates 1a & 1b) showed it to have a surface textured in a cellular pattern characteristic of Bénard Cells [9]. This effect, caused by retained solvent (see Discussion), effectively separates the paint and barrier coating except along the Bénard Cell boundaries. It was found that relatively long paint cure periods (6 days at 25 °C) were required to avoid Bénard Cell formation and the adhesion problems associated with it. Similar adhesive failures were observed in lap shear tests when the paint had short cure times.

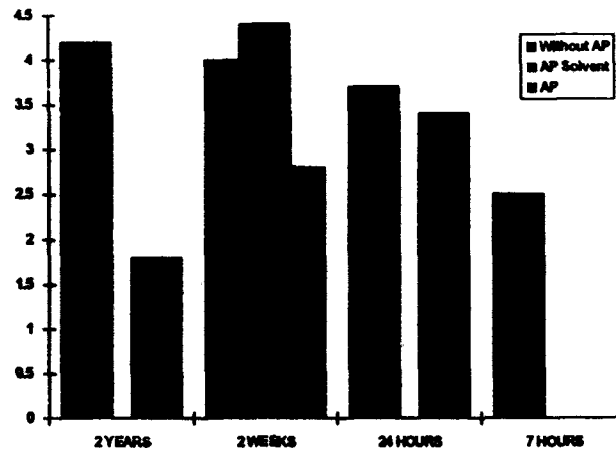**Lap Shear Strength (kN)**

Figure 2: *Lap shear strength of PR 1750/Barrier coating/Paint system vs Paint Age.*
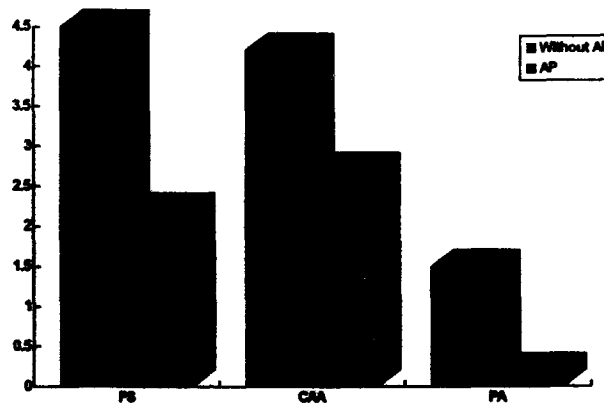
Lap Shear Strength (kN)

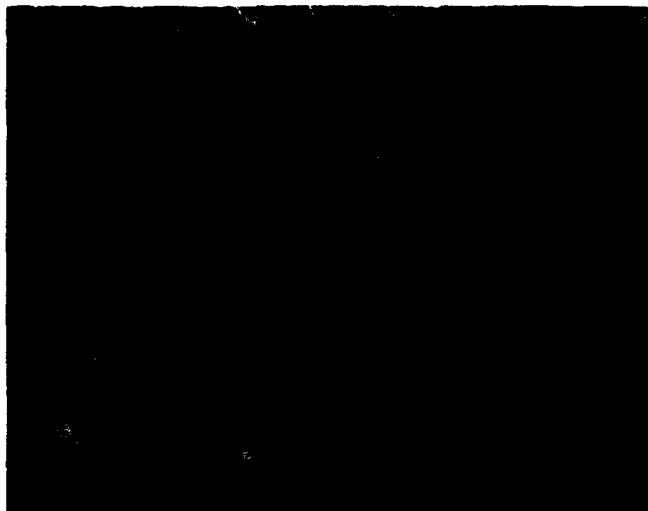Figure 3: *Effect of Adhesion Promoter on Lap Shear Strength of PR 1750/Barrier coating/Paint system.*

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Table 3: Peel Strength* of Immersed Sealant System

Barrier Coat	Adhesion Promoter	Weeks Immersed	2 years** Strength*	2 years** Failure	2months** Strength	2months** Failure	2 weeks** Strength	2 weeks** Failure	48hours** Strength	48hours** Failure	7 hours** Strength	7 hours** Failure
Pres Syst.	W/O	1	20#	100%C	20#	100%C	19.5	30%C	10.5	100%A	6.1	95%A
	W/O	3	20#	100%C	20#	100%C		80%C	17.5	90%C	5.2	95%A
	APS	1	20#	100%C								
	APS	3	20#	100%C								
	AP	1	7.9	20%C					7.6	100%A		
	AP	3	10.5	70%C					7.6	70%C		
Poly amide	W/O	1	20#	100%C	10.5	30%C	3.5	90%A	2.5	92%A	2.6	100%A
	W/O	3	20#	100%C	6.9	30%C	3.8	50%A	1.7	96%	2.6	100%A
	APS	1	20#	100%C								
	APS	3	20#	100%C								
	AP	1	NR	100%A					3.4	80%A		
	AP	3	NR	100%A					2.5	92%A		
CAA	W/O	1	20#	100%C	NR	60%C	4.4	90%A	2.5	97%A	0.9	97%A
	W/O	3	20#	100%C	NR	80%C	5.2	98%A	1.7	93%A	1.0	100%A
	APS	1	20#	100%C								
	APS	3	20#	100%C								
	AP	1	6.1	100%A					4.4	100%A		
	AP	3	6.1	100%A					3.5	92%A		
PU	W/O	1	4.4	100%AD	3.1	100%AD	6.9	90%AD	4.4	83%AD	2.6	100%AD
	W/O	3	4.4	100%AD	1	92%AD	6.9	100%AD	5.2	100%AD	1.3	100%AD
	APS	1	4.4	100%AD								
	APS	3	4.4	100%AD								
	AP	1	4.4	100%AD				35	100%AD			
	AP	3	4.4	100%AD				27.5	100%AD			

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Table 3 (Contd): Peel Strength* of Immersed Sealant System

Barrier Coat	Adhesion Promoter	Weeks Immersed	2 years** Strength*	2 years** Failure	2months** Strength	2months** Failure	2 weeks** Strength	2 weeks** Failure	48hours** Strength	48hours** Failure	7 hours** Strength	7 hours** Failure
BC	W/O	1	4.8	100%A	5.2	100%A	0.3 100%A	2.6	10.2	100%A		85%A
	W/O	3	2.6	100%A	6.8	100%A	0.7 100%A	12.7	10.2	100%A		85%A
EP/PS	APS	1	4.4	100%A								
	APS	3	3.8	100%A								
	AP	1	2.6	100%A					6.8	92%A		
	AP	3	2.6	100%A					3.1	100%A		

BC = Barrier Coating
AP = Adhesion Promoter
APS = Adhesion Promoter Solvent
W/O = Without Adhesion Promoter

the cohesive failure traces are unstable and the values shown are estimated averages

**2 Years = Paint Age in Years prior to Barrier Coating Application
**2 Months = Paint Age in Months
**2 Weeks = Paint Age in Weeks
Weeks Immersed = Weeks immersed in Avtur fuel

*Lap Shear Strengths measured in megapascals (MPa)
%A = % adhesion failure between paint and barrier coating
%AD = % adhesion failure between sealant and barrier coating
%C = % cohesive failure in the sealant

NR = not recorded

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Table 4: Lap Shear Strength* of Immersed Sealant System

Barrier Coating	AP	Weeks Immersed	2 years** Strength	2 years** Failure	2 weeks* Strength	2 weeks* Failure	24 hours** Strength	24 hours** Failure	7 hours** Strength	7 hours** Failure	Accel. Cure Strength	Accel. Cure Failure
Pres Syst.	W/O	1	12.7	100%C	12.5	90%C	11.4	95%C	7.6	40%C	13.8	95%C
		3	13.1	90%C	13.1	100%C	11.7	80%C	7.6	40%C		
	APS	1			13.7	100%C	12.6	80%C			6.8	45%C
	AP	1	4.4	95%A	7.2	75%A	11.8	80%C				
		3	5.5	95%A	8.8	70%C						
Poly amide	W/O	1	0.8	100%B	3.4	50%A	3.7	85%A	4.7	70%A	4.5	75%A
		3	1.6	95%B	4.9	50%A	3.6	90%A	5.8	50%C		
	AP	1	@	@			4.5	95%A			0.7	100%A
		3					4.8	95%A				
CAA	W/O	1	9.6	90%C	8.8	100%C	4.7	60%A	2.9	90%A	13.0	95%C
		3	9.7	70%C	9.3	100%C	4.8	70%A	3.8	100%CB		
	AP	1									8.5	55%A
PU	W/O	1	0.2	100%AD	4.7	90%C	6.5	100%C	7.6	70%A		
		3	0.7	70%AD	3.6	60%C	9.0	85%C	7.3	70%C		
	AP	1	0.04	100%A			7.8					
		3		100%A			6.5					
EP/PS	W/O	1	@	100%C	2.1	100%C	3.6	50%C	0.9	100%CB		
		3	@	100%C	1.9	100%C	3.7	30%C	3.2	90%CB		
	AP	1	0	100%A			3.1	95%A				
		3	0	100%A			2.2	98%A				

Lap Shear Strength is measured in megapascals (MPa)

Table 4 (Contd): Lap Shear Strength* of Immersed Sealant System

BC	=	Barrier Coating	
AP	=	Adhesion Promoter	
APS	=	Adhesion Promoter Solvent	
W/O	=	Without Adhesion Promoter	

%A	=	% adhesion failure between paint and barrier coating
%C	=	% cohesion failure in the barrier coating
%CB	=	% cohesion failure in the barrier coating

NR = not recorded

@ broke before tested

**2 Years	=	Paint Age in Years prior to Barrier Coating Application
**2 Months	=	Paint Age in Months
**2 Weeks	=	Paint Age in Weeks
Weeks Immersed	=	Weeks immersed in Avtur fuel

4.2.2 Effect of Adhesion Promoter on Adhesion

Examination by both peel and lap shear tests showed that the adhesion of the barrier coating to well cured paints (two years, two weeks, accelerated cure) was greatly reduced when adhesion promoter was applied to the paint. Paints treated with adhesion promoter generally displayed weak adhesive failure between the paint and the barrier coating (Tables 3 & 4; Figure 3).

Systems in which paints had only short cure times generally failed by adhesive separation between the paint and the barrier coating whether or not they had been treated with adhesion promoter.

4.2.3 Effect of Barrier Coating Composition on Adhesion

Of the barrier coating compositions examined, the current system was consistently found to have the best adhesion to paint. Where the paint had been fully cured (two years, two weeks, accelerated cure) the cycloaliphatic amine barrier coating was also found to have good adhesion to the paint. Both the polyamide and the polyurethane displayed reasonable adhesion to fully cured paint although, surprisingly, the polyurethane barrier coating failed adhesively between the sealant and itself. This was the only well cured system to fail in this manner. The epoxy/polysulfide had poor adhesion to the paint (Tables 3 & 4; Figure 4).
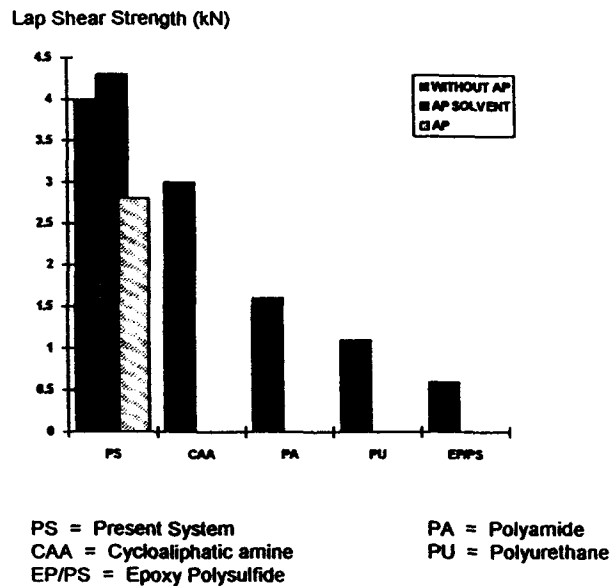
Lap Shear Strength (kN)

PS = Present System
CAA = Cycloaliphatic amine
EP/PS = Epoxy Polysulfide

PA = Polyamide
PU = Polyurethane

Figure 4: Lap shear strength vs. Barrier coating composition.

With reduced paint cure times (7 hours, 24 hours, 48 hours), the adhesion of the cycloaliphatic amine barrier coating decreased significantly, giving, for instance, only fair adhesion on paint cured for 7 hours. The next strongest adhesion produced after short paint cure time was the polyurethane barrier coating which improved with decreasing paint cure time. The polyamide system also exhibited reasonable adhesion, however the epoxy/polysulfide barrier coating was again the weakest of those tested.

## 4.2.4 Effect of Fuel Immersion Times on Swelling and Adhesion

The paints and sealants were not significantly swollen on immersion in Avtur (F35) fuel while the cycloaliphatic amine, polyurethane and polyamide barrier coatings also exhibited minimal swelling. Significant swelling (as determined by weight increase) was observed, however, with the present barrier coating and the epoxy/polysulfide barrier coatings, showing weight increases of 1.4% and 1.1% respectively after three weeks (Table 5). When examined by volume swell in Avtur, the increases in the present barrier coating after two, three and 35.5 weeks were 1.4%, 2.06% and 6.6% respectively. The changes in volume swell in F34 RAAF fuel (avtur containing the antiicing inhibitor diethylene glycol methyl ether (DGME) at 0.15%) were 1.7%, 2.5% and 7.1% respectively (Figure 5).

Table 5(A): Weight increase on immersion in Avtur

Barrier Coating	Sealant Present	Weight before Immersion	Weight after One Week	Weight after Two Weeks
Paint Only	No	17.52	17.52	17.53
Present System (PS)	No	19.93	20.13	20.20
Present System (PS)	Yes	46.09	46.28	46.27
Polyamide (PA)	No	18.79	18.83	18.85
Polyamide (PA)	Yes	48.16	48.22	48.17
Polyurethane (PU)	No	18.56	18.60	18.60
Polyurethane (PU)	Yes	49.50	49.63	49.55
Cycloaliphatic Amine (CAA)	No	18.84	18.86	18.87
Cycloaliphatic Amine (CAA)	Yes	42.00	42.10	42.03
Epoxy Polysulfide	No	19.27	19.44	19.48
Epoxy Polysulfide	Yes	39.61	39.72	39.68

Table 5(B): Percentage volume swell of barrier coating in aviation fuel

Immersion Time (Weeks)	Avtur/DGME	Avtur
2	1.7	1.4
3	2.5	2.0
35.5	7.2	6.6
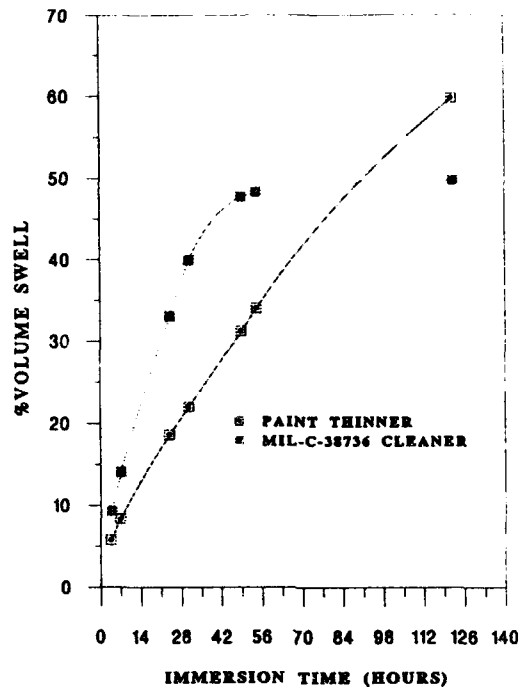
Figure 5: % Volume swell of Barrier 3580.

The swelling exhibited by barrier coatings in Avtur was eliminated or reduced when overcoated by the polysulfide sealant. Longer tests may be needed to gauge this effect. An investigation of the volume swell of the barrier coating in the paint thinner (020-707) used for the Integral Fuel Tank Coating showed it to have a greater, albeit slower, effect on swelling than did the MIL-C-38736 cleaning solvent (Figure 3).

No difference in the adhesion of the complete sealant system was observed after one and three weeks immersion in F35 fuel.

4.2.5 Effect of Aging of the Barrier Coating

When aged 3M EC2216 barrier coatings were examined, adhesion was reduced compared with adhesion of fresh samples. Microscopy of the aged coatings showed the presence of voids (Plates 2a). Sectioning of the sample for microscopy

led to separation of the sealant and barrier coating (Plate 2b). Analysis of the water content of the barrier coatings by the Dean and Stark method showed that the aged sample had higher water content which may have contributed to the poor adhesion. In view of these results, aged barrier coatings were discarded. Water contents were found to be:

Batch 1 (aged sample - 2 years)		Batch 2 (new sample)	
EC 2216A	1.2%	EC 3598A	0.2%
EC 2216B	0.2%	EC 3598B	0.3%

5. *Discussion*

A number of authors have investigated sealant failures characterised by separation at the paint/sealant interface. Usmani et al. [10] reported that the sealant/coating combination works well initially and there is apparently adequate adhesion of the polysulphide rubber to the polyurethane coating. Upon extended aircraft usage, however, leaks do develop which cannot be repaired by removing the old polysulfide rubber and reapplying fresh sealant because of insufficient adhesion. Usmani [10] also found that heat treatment of polyurethane paint at 121°C produced a surface to which the polysulfide would not stick and that the adhesion of the polysulfide sealants slowly deteriorated with age of the paint [11-13]. They correlated the loss of adhesion with the loss of unreacted isocyanate groups on the polyurethane surface, disappearance of these groups being accelerated by increased temperature and humidity. In a similar study on the effect of aging of epoxy priming paints on sealant adhesion, Wilford and Day [14] found that adhesion of one of the epoxy paints decreased significantly with aging of the primer but three other 'tolerant' primers were unaffected. The cause of this difference was not determined.

Adhesive failures associated with the overpainting of aged epoxy paints are well known in the paint industry even at drying times of less than 24 hours. Prior to the present investigation, excessive aging of the primer was considered a possible cause for the sealant failures plaguing the RAAF F111 aircraft. However, the finding of the opposite phenomenon, i.e. good adhesion of the barrier coating to aged paints but poor adhesion to freshly cured paints stimulated further work to determine the cause of the adhesive failures in the F111 tanks.

Plate 2a: *Void formation in aged barrier coating.*

Plate 2b: *Separation of Paint and Epoxy Barrier Coating (Aged).*

The long paint cure times needed to achieve good adhesion (Table 6) were surprising given the film thickness (ca 20-25 μm). The slow evaporation of cyclohexanone from the paint solvent has been reported to require an extensive period of time [15]. Furthermore, isocyanates can also complex with ketones to form ketimines which will further slow solvent evaporation. Where the presence of ketimines is suspected, the application of heat is often used to assist paint cure by driving off the ketone produced from ketimine breakdown.

Table 6:   Results of Peel Strength Adhesion

					Paint Cure Times (hours)										
Temp C	15	17	19	20	24	25	35	40	44	48	50	54	72	96	120	144
25 C	F	F	F	F	F	F	F	F	F	F	F	F	F	F	F/P	P
37 C	F	F	F	F	F	F	F	F/P	P	P	P	P	P	P	P	P
40 C	F	F	F	F	F	F/P	P	P	P	P	P	P	P	P	P	P
50 C	F	F	F	F	F/P	P	P	P	P	P	P	P	P	P	P	P
60 C	F/P	P	P	P	P	P	P	P	P	P	P	P	P	P	P	P

F = Fail   P = Pass

Adhesive separation = failure (i.e. when adhesive strength is less than 20 N/mm at a cross head speed of 50 mm/min).
Cohesive separation = pass (i.e. when adhesive strength is greater than 20 N/mm).

The cause of the poor adhesion in the sealing system became apparent with the observation of Bénard Cell formation under the epoxy barrier coating. Bénard Cells are normally formed on the surface of a paint film by currents set up as a result of solvent circulation. When the solvent volatilises at the surface of a wet film, solvent in the body of the film migrates to the surface to replenish the surface supply. To facilitate solvent escape, small pores form which terminate at the surface of the film. These pores commonly result in the formation of geometric patterns observable on the surface of the film as the paint being emitted from these eruptions spreads away from the pore to the outside of this crater [9]. Boundaries are formed between adjacent pores resulting in the typical cellular structure seen in Plate 1a and 1b. However, in the present case, a solvent mixture is being released to the surface of a paint film overcoated with a thick, epoxy polyamide barrier coating. Curing epoxy polyamides contain long fatty acyl components which are susceptible to solvent effects such as the formation of Bénard Cells. Somewhat unusual, however, is the fact that the Bénard Cells are formed on the mating or mirror surface to the paint from which the solvent is leaving.

In an effort to overcome the poor performance of fuel tank repairs, General Dynamics [16] conducted a study on improving adhesion between sealant and paint by the use of adhesion promoters. This study, by Carroll and Pritchard [16] found that the adhesion of paint to sealant was improved by some adhesion promoters but not others. For systems where the adhesion promoter was completely ineffective, abrasion was required to provide a mechanical key for improved adhesion. Usmani et al [10] similarly found increased bond strength

with an adhesion promoter (an alkyl phosphatotitanate) although their observation that a solvent wipe removed the adhesion promoter from the paint is surprising in that it precludes chemical bonding which is the basis of action of the adhesion promoters.

Application of an epoxy barrier coating between the paint and the sealant for F111 aircraft was reported in a study by General Dynamics [17]. The barrier coating originally chosen was 3M's EC-2216, however this was later changed in service to 3M's EC-3598. The adoption of a barrier coating appears courageous in view of a number of unresolved problems described in the report. These problems included loss of adhesion of the sealant to the epoxy barrier coating with time and temperature, loss of flexibility on exposure to low temperatures (cracks in the barrier coating), as well as loss in hardness, loss in flexibility and increased swelling on fuel immersion. The authors obviously placed great faith in the polysulfide overlay to alleviate many of these problems suggesting that for actual fuel tank applications, however, the epoxy will be covered by a thick fillet of polysulfide sealant which should protect the epoxy from the fuel [17].

The results of the fuel immersion tests in the present investigation support the General Dynamics contention that the use of polysulfide reduces swelling in fuel. However, care must be taken with results from short term trials with sealants. A case in point is that at one time, USAF used sealants in fuel tanks without an underlying priming paint. They did this on the basis of laboratory evaluations showing that the sealant alone would protect the tanks from corrosion. They based this on short term exposure results [18]. In service, however, the onset of corrosion occurred several months later.

Examination of the effect of barrier coating composition on fuel tank sealant performance supports the barrier coating selected by General Dynamics. Although the present barrier coating was swollen by fuel, it consistently exhibited the best bond strength to priming paint of all candidate materials. Evaluation of a polyurethane as a barrier coating, showed it to have moderate adhesion to the priming paint but poor adhesion to the sealant. On the other hand, it exhibited the best adhesion to freshly cured paint, presumably by interaction of the isocyanates with amines and hydroxyls in the barrier coating. The epoxy polysulfide consistently exhibited poor adhesion.

The adhesion of sealant systems using barrier coating in combination with the adhesion promoter, as used in the F111 fuselage tanks, was never examined; it is therefore not surprising that adhesion failures occurred in service. The present report has shown that this combination results in poor adhesion and that removing the adhesion promoter significantly improves the durability and strength of the sealant system. Likewise, paint cure has a significant affect on the integrity of the barrier coating and its adhesion to the priming paint. By modifying these two procedures, a system for sealing F111 fuselage fuel tanks is achieved which has adhesion greater than the cohesive strength of the polysulfide.

6. Operation of the Second Deseal/Reseal Program

Although the changed deseal/reseal procedures were largely finalised prior to desealing the first F111 aircraft, the onset of the resealing program in the aircraft required that the range of conditions under which procedures could be employed

be gradually expanded. This expansion was required to meet material curing requirements under the environmental conditions prevailing at RAAF Amberley. The evolution in procedures [19-20] is contained in MRL and RAAF minutes and is instructive for operational considerations.

When the paint cure requirements were originally investigated prior to the deseal/reseal programme commencing, it became apparent that good adhesion between the paint and the barrier coating required paint cure times considerably longer than those requiring the paint to meet the solvent resistance specification which existed up to that time. It also became apparent that a minimum temperature requirement was needed for adequate paint cure.

When resealing of the first two F111 aircraft commenced at Amberley, temperature control equipment was unavailable at the overhaul facility. As a result, the contractor undertook resealing of these two aircraft using an ambient temperature cure regime with relatively long cure periods. However, the temperature at the facility fell well below the minimum temperature requirement at night. As such, it was impossible to determine the elapsed total cure time above the minimum required temperature.

As a result of installation of heating capacity at the overhaul facility, a considerable range of temperature regimes became available for the reseal of F111's after aircraft No 2. MRL was therefore tasked by RAAF with determining whether the IFTC overcoat curing temperature range could be expanded from as low as 20 °C up to 50 °C and on whether the cure could be accelerated under high temperature up to the point of initial fuel/MEK resistance followed by reducing the temperature to final cure. RAAF also requested that MRL examine the possibility of producing a table of cure times to achieve final cure. [21].

Following further evaluation, the conditions of cure were then expanded to accelerate the cure of IFTC by the use of high temperatures with a restriction for letting the paint flash dry for 30 minutes after application. It was found that it was possible to accelerate the IFTC cure under high temperature and subsequently reduce temperature, or conversely gradually increase temperatures during cure. However, it was found that it was not permissible to partially cure the paint at high temperature, apply the barrier coating and allow it to cure. This procedure did not develop maximum adhesion between the barrier coating and the paint [22].

In view of the quick overhaul times achievable using elevated temperatures, RAAF further requested that paint cure schedules up to 60 °C be considered [23]. Following further work, time schedules were compiled at temperatures up to 60°C (Table 6) which met the paint cure requirements [24]. Because of daytime temperature variations, this tabulation was subsequently converted to a graph of so-called `heat units' (Figure 6) achievable at various temperature-time schedules, a heat unit being defined as the percentage cure gained per hour at a specific temperature, e.g. cure is achievable in 144 hours at 25 °C. Therefore, heat units per hour at 25 °C $= 1/144 \times 100 = 0.69\%$ [25]. These units are additive so that if the temperature cycle of an aircraft is known, then the additional time required to achieve acceptable cure can be calculated. Once full cure is achieved (100%), the integral fuel tank paint could be overcoated with barrier coating.
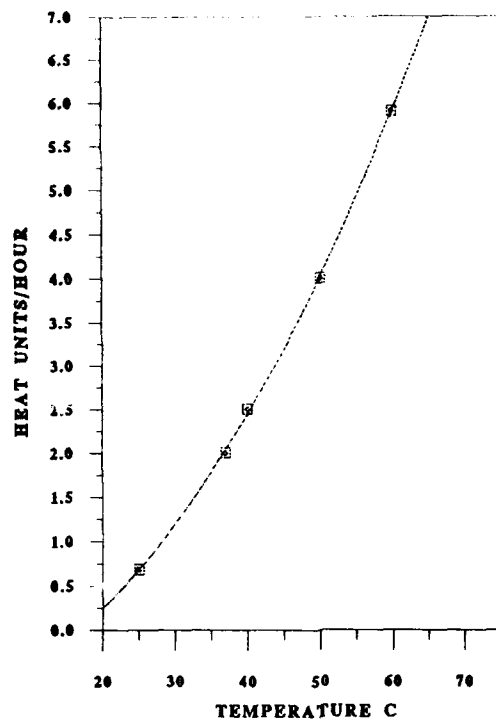
MINIMUM CURE TIMES

FOR INTEGRAL FUEL TANK COATING (PAINT)

Figure 6: Minimum cure times for integral fuel tank coating (paint).

7. Conclusions

From this study, the following conclusions have been drawn:

(i) Sealant performance in the fuselage fuel tanks of F111 aircraft was critically dependent on the adhesion of the barrier coating and the sealant applied over it, to the priming paint used to protect the tank surfaces. Two factors were found to critically affect the adhesion of the barrier coating and sealant to this priming paint. These factors were (a) the requirement for adequate cure of the priming paint prior to application of the barrier coating and sealant, and (b) the use of an adhesion promoter between the paint and the barrier coating.

(a) An increased degree of cure of the priming paint is needed beyond the former requirement of resistance by the paint to methyl ethyl ketone. The paint contains the low volatility solvent cyclohexanone which requires extended cure. Failure to carry out proper cure results in solvent attack on the underside of the barrier coating and poor adhesion between the paint and the barrier coating.

(b) The use of the recommended adhesion promoter between the paint and barrier coating was found to lead to greatly reduced performance by the fuel tank sealant system. The adhesion promoter was found to reduce the adhesive strength between the paint and barrier coating.

(ii) The present barrier coating is easily the best performed of those tested.

From the above it is recommended that for maximum adhesion of the barrier coating system, the present barrier coating should be retained and applied over well cured priming paint. The painted surface should be solvent cleaned, however the titanate based adhesion promoter should not be employed.

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TITLE

Improved Resealing Procedures for the Second Deseal/Reseal Program in RAAF F111 Aircraft Fuel Tanks

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Curing	Bonding	Barrier coatings

ABSTRACT

A high temperature-resistant polyester sealant was originally employed to seal faying surface grooves in F111 fuselage fuel tanks as well as structural voids and aerodynamic surfaces. Chemical hydrolysis of this material was detected in March 1974 and led to its replacement by a polysulfide sealant in the late 70's and early 80's. Where possible, the polyester was removed, however much of it remained inaccessibly located between the overlapping panels. It was found that the continued hydrolysis of this residual polyester generated sufficient pressure to penetrate the polysulfide sealant resulting in fuel leaks as well as appearing externally in trails along the skin of the aircraft. To protect the polysulfide from penetration, a mechanical barrier was applied in the form of a layer of epoxy polyamide between the polysulfide and the polyester. This system was successful for some years, however the occurrence of fuel leaks had become unacceptable by 1987. All F111 aircraft suffered numerous fuel leaks over the period 1988-91 including a number of safety-of-flight (SOF) incidents. A detailed investigation was therefore requested to identify the cause(s) of sealant failures. This investigation found that the sealant and the underlying epoxy barrier coating could be manually peeled from the painted tank surface when prepared by the recommended methods and that two procedures were responsible for the poor adhesion. One of the procedures was found to lead to incomplete cure of the underlying priming paint resulting in solvent attack of the barrier coating. The second procedure, involving the use of a so-called "titanate adhesion promoter", significantly reduced peel strength of the barrier coating to the paint. Modification of these two steps resulted in adhesive bond strengths greater than the cohesive strength of the polysulfide sealant. These changes were adopted for the second deseal/reseal program of the F111 aircraft fleet. Following teething problems with the first aircraft, the procedures were improved and the F111 fleet remains leak-free after two years.

Improved Resealing Procedures for the Second Deseal/Reseal
Program in RAAF F111 Aircraft Fuel Tanks

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